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Harvesting Sustainability: Transforming Rice Straw Wastes into Eco-Friendly Silica Gel

Ahmad Said[a,*], Aprisilia Risky Wijaya[b], Gazali Majid[a], Dede Ulfah[a], Ramadhani[c], Muhammad Miqdam Musawwa[d]

[a] Sekolah Tinggi Teknologi Cipasung, Tasikmalaya, West Java 46466, Indonesia
[b] Diploma Program of Analytical Chemistry, Department of Chemistry, Universitas Islam Indonesia, Yogyakarta 55584, Indonesia
[c] Department of Environmental Engineering, Faculty of Engineering, King Mongkut's University of Technology Thonburi, Thailand.
[d] Department of Chemistry, Universitas Islam Indonesia, Yogyakarta 55584, Indonesia
*Corresponding author: ahmadsaid@sttcipasung.ac.id

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ABSTRACT

The vast quantities of rice straw residue generated every year present both a challenge and an opportunity. To address the challenge, this work took the opportunity by transforming rice straw waste into eco-friendly silica gel. Synthesis of rice straw-based silica gel was via sol-gel method with the calcination temperature of 700 °C for 2 h and the addition of sodium hydroxide 2 molar. Characteristics of silica gel and its raw material have been investigated using several techniques such as FTIR, XRD, SEM, and BET surface area. The synthesized silica gel showed obvious peaks of Si-O-Si and O-H, aggregated smaller particles, crystal nature, and high surface area. This innovative work contributes to agricultural waste management and the circular economy for a sustainable future.

Keywords: Biomass, agricultural waste, rice straw, silica gel, sustainability growth

ABSTRAK

Meningkatnya kuantitatas residu jerami padi yang dihasilkan setiap tahun menghadirkan tantangan sekaligus peluang. Untuk mengambil kesempatan sekaligus menghadapi tantangan tersebut, penelitian ini melakukan inovasi dengan mengubah limbah jerami padi menjadi gel silika yang ramah lingkungan. Sintesis gel silika berbasis jerami padi dilakukan melalui metode sol-gel dengan suhu kalsinasi 700 °C selama 2 jam dan penambahan natrium hidroksida 2 molar. Karakteristik silika gel dan bahan bakunya telah diteliti menggunakan beberapa teknik seperti FTIR, XRD, SEM, dan luas permukaan BET. Silika gel yang disintesis menunjukkan puncak Si-O-Si dan O-H yang jelas, partikel agregat yang lebih kecil, sifat kristal, dan luas permukaan yang tinggi. Karya inovatif ini dapat berkontribusi pada pengelolaan limbah pertanian dan ekonomi sirkular untuk masa depan yang keberlanjutan.

Kata kunci: biomassa, limbah pertanian, jerami padi, gel silika, sustainability growth

INTRODUCTION

One of the byproducts of rice cultivation that still leaves problems in agriculture is rice straw. Every harvest season, there is rice straw amounting to 45% of the total rice crop (Lu & Hsieh, 2012). This figure exceeds the amount of rice husk waste obtained, which is 20% (Abellan-Garcia et al., 2023). According to the report from Statistics of Sleman Regency, the average

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rice production in Mlati District, Sleman Regency is 58.36 quintals/hectare, which means that there are approximately 26.26 quintals of rice straw produced by Mlati District farmers every 1 hectare (BPS, 2019).

Various treatment techniques of rice straw residue in Indonesia have been widely carried out. For example, rice straw is transformed into creative products and works of art (Dharsono et al., 2019; Pandu et al., 2021), as an animal feed ingredient (Sugama & Budiari, 2012), as a planting medium (Herdiana, 2020; Nurmalasari et al., 2021), as a substitute raw material for producing ethanol (Karisma, 2015; Winarsih, 2013), activated carbon (Goodman, 2020; Sudhan et al., 2017), and silica-based materials (Bhattacharya & Mandal, 2018; Yekeen et al., 2023). Among those utilizations, transformation of rice straw into silica-based materials has been recently paying attention to the sustainability due to its abundance and value-added property.

Ekwenna et al. (2023) have prepared bio-based silica from rice straw leached with cassava-steep wastewater. The study found amorphous and crystalline silica materials under methanogenic and acidogenic conditions, respectively (Ekwenna et al., 2023). Other researchers have studied the rice straw-based silica removal until 92% using ammonia (Pal et al., 2022).

Rice straw-based silica materials are potentially applied for adsorption such as reduction of nitrate (Robles-Jimarez et al., 2022), removal of mercury (Niu et al., 2014), fluoride removal (Pillai et al., 2020), ferrous metal adsorption (Rusdianasari et al., 2020). Silica particles derived from rice straw can be obtained through several steps: pretreatment (washing and drying), extraction of silica, and formation of gel (Bhattacharya & Mandal, 2018; Motlagh et al., 2020). Chemical agents commonly used to extract silica from rice straw are K_2CO_3, $KHCO_3$, Na_2CO_3, $NaHCO_3$, NaOH, and KOH.

The objective of this research is to utilize rice straw wastes obtained from the rice field in Mlati District, Sleman Regency, and demonstrate a synthesis of silica gel from rice straw wastes via sol-gel method. The obtained rice straw wastes were calcined under a furnace at 700^{o}C because silica content was highest at 700^{o}C of calcination compared to 300^{o}C and 500^{o}C (Said et al., 2014). Sodium hydroxide solution was used in this study as the basic chemical agent to form sodium silicate which is soluble in water. The utilization of rice straw to produce silica gel is proposed to address the problem of rice straw waste management and reduce the environmental

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impact associated with conventional manufacturing processes of silica gel.

METHODS

2.1. Materials

Rice straw waste was collected from a farmer's rice field in Jaten Village, Mlati District, Sleman Regency. Sodium hydroxide (NaOH) 2 M, hydrochloric acid (HCl) 1 M, and distilled water were used in this experiment.

2.2. Synthesis of Silica Gel

Rice straw waste (RSW), at first, was washed with detergent and rinsed using tap water to withdraw dust or sludges. Lastly, RSW was cleaned with distilled water. Drying of RSW was conducted in oven at 105^{o}C for 4 hours. Prior to calcination, the RSW was cut into smaller pieces ($\pm$ 1 cm). To form ash, the RSW was calcined in a furnace at 700^{o}C for 2 hours. The obtained ash was called rice straw ash (RSA) and cooled in a desiccator.

The amount of RSA (3 grams) was dissolved into 40 mL of NaOH 2 M to extract silica particles. RSA solution was stirred along 24 hours and then filtered using a cellulose filter paper (Whatman ashless grade 42). The pH of the obtained solution was adjusted to 8 using HCl 1 M. To produce silica gel (SG), the solution aging was needed for 48 hours.

2.3. Characterization of Silica Gel

The properties of RSA and SG were examined using instrumental analyses such as FTIR (Nicolet Avatar 360 IR), SEM (JSM-6510LA), XRD (Bruker AXS D8 Advance), and BET surface area analyzer (NOVA 1200e). The analyses were conducted to study the functional groups, morphology, crystallinity, surface area, and porosity of materials.

RESULTS AND DISCUSSION

Based on the goals to be achieved, RSW obtained from farmers' fields in Jaten Village, Mlati District, Sleman Regency, can be converted into silica gel by sol-gel method in this experiment. Washing RSW with detergent can eliminate organic and inorganic contaminations derived from soil due to polar and nonpolar components in detergent (Moraes, 2016). Washing with distilled water is to remove the remnants of inorganic impurities that may be left behind from rice straw. Drying RSW in oven is to remove water content so that the calcination occurs evenly. Calcination of RSW at 700^{o}C for 2 hours resulted in bright white powder which shows the perfection of burning, no carbon left (**Figure 1**). The product is called rice straw ash (RSA).

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Figure 1. RSA from calcination at 700°C

The calcination process of rice straw aims to form silica and remove other unwanted organic compounds. Organic compounds such as carbon will decrease at high temperatures and result in H_2O and CO_2 gases. At that temperature, it is expected that the silica content produced is very high, as happened in the calcination of rice husks at 700°C, the silica content is 87% (Hindryawati & Alimuddin, 2010; Said et al., 2014).

The as-prepared RSA is formed into a gel through the formation of sodium silicate salts (Na_2SiO_3). The rice straw ash is dissolved into NaOH 2 M and stirred for 48 hours which serves to bind silica from RSA strongly, thus forming a thickened liquid. To remove insoluble substances, the liquid is filtered which can separate ash. The resulting filtrate is called sodium silicate solution.

Sodium silicate solution is a precursor to gel formation. Addition of HCl can drop the pH of sodium silicate from 14 to 8. Hydrochloric acid has a high affinity level so it can be used to lower the acidity level of gel solutions. Acids that react with sodium silicate will form ortho silicic acid which is an intermediate product of the hydrolysis process (Ali & Asghar, 2010). This product is very unstable and will change suddenly to form silica sols due to condensation (release of water molecules). Excessive condensation makes the silica sols turn into silica gel (see the mechanism in **Figure 2**).

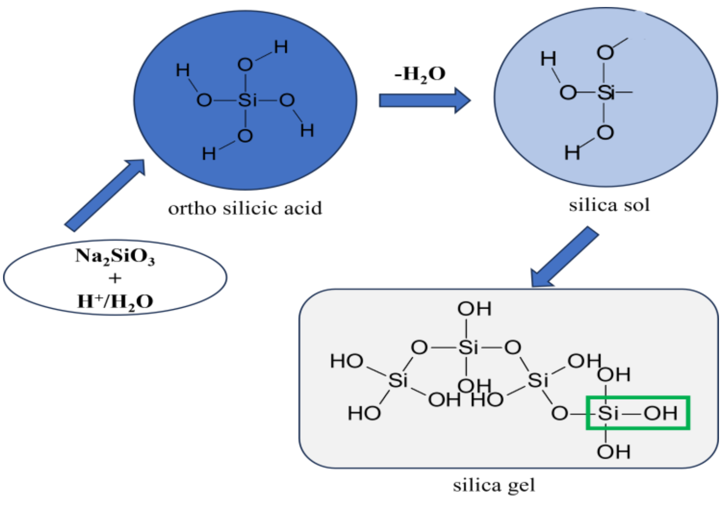

Figure 2. Mechanism of silica gel formation

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In the synthesis of silica gel from rice straw, it is expected that there are active compounds: siloxane (Si–O–Si) and silanol (Si–OH). The presence of these two groups leads silica gel to be beneficial for absorbent, desiccant, and catalyst.

3.3. Characteristics of Silica Gel

The synthesized silica gels have been characterized by FTIR, SEM-EDS, XRD Spectroscopy, and BET surface area techniques. Characteristics of RSA is also given here as starting material of silica gel.

FTIR analysis is used to determine the functional groups present in RSA and SG through the interpretation of molecular vibrations. Active groups of silica gel and the starting material in the forms of siloxanes (Si-O-Si) and silanol (Si-OH) are presented in the FTIR spectra (**Figure 3**).

The presence of external symmetrical stretching of Si-O as part of the siloxanes appears at 794.7 cm^{-1}, while the bending bond occurs at 470.6 cm^{-1}. A specific peak at 1095.6 cm^{-1} indicates internal asymmetric stretching for the Si-O bond of Si-O-Si, the broader peak is shown in the spectrum of SG compared to RSA which is attributed to the successfully obtained silica gel. Meanwhile, the silanol functional group appears at 1635.6 cm^{-1} as the bending vibration of the OH group of Si-OH. A wide peak at about 3400 cm^{-1} (3448.7 cm^{-1}) indicates the characteristics of the hydroxyl group. The absence of a peak at 960 cm^{-1} indicating the Si-O stretching bond of Si-OH group is the reason that some silanol groups have turned into siloxanes due to condensation at high temperatures.

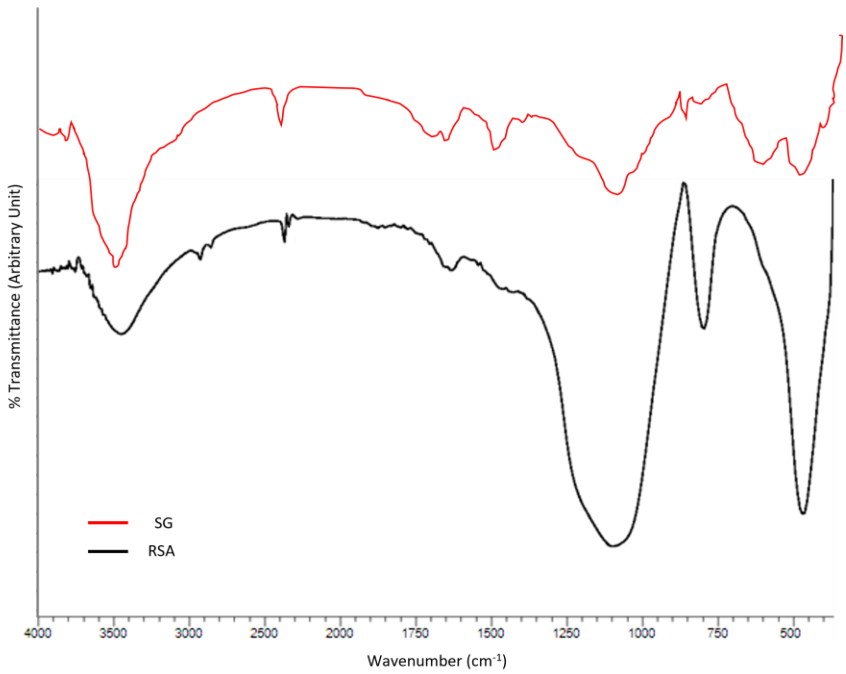

Figure 3. FTIR spectrum of RSA and SG

Crystallinity of silica gel can be investigated using XRD spectroscopy. The presence of peak at 2θ = 21.66 deg (**Figure 4**) shows amorphous silica of RSA with the diameter of 4.09 angstrom. This result is similar to the previous literature (Khorsand et al., 2013). The crystalline properties of silica gel synthesized from rice straw ash are evident on the XRD chart which has a high intensity at 2θ = 31.71; 45,26; and 56.37 deg (Figure 4). The nature of these crystals becomes a distinctive property of silica gel.

Morphological analysis of silica gel using SEM microscopy provides different morphological characteristics between rice straw ash and silica gel (Figure 5). Rice straw ash has an uneven morphology or is shapeless (amorphous) but has pores that can be utilized. Meanwhile, silica gel represented aggregated smaller particles, this clearly indicates the potential product was silica.

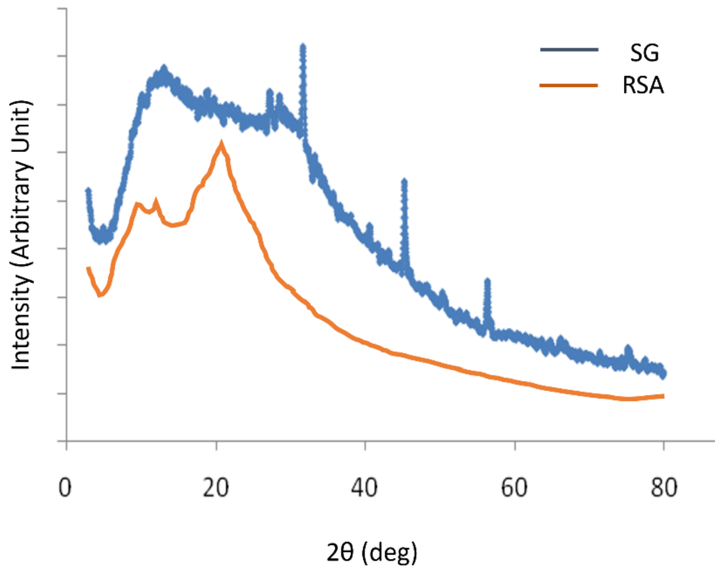

Figure 4. XRD Spectrum of RSA and SG

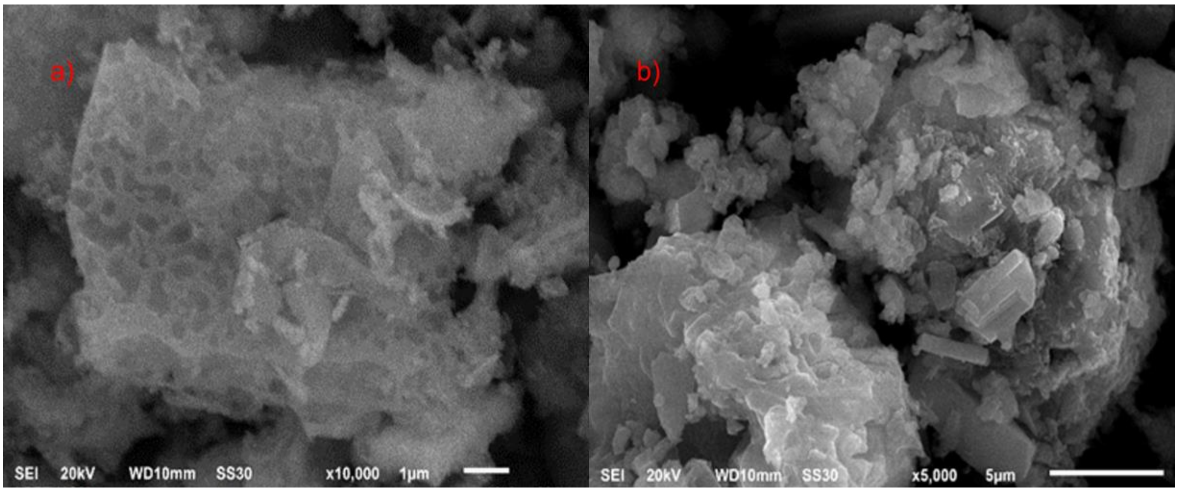

Figure 5. SEM morphology of RSA (a) and SG (b)

Table 1. BET Surface Area Analysis Results

Parameters	RSA	SG
Surface area ($m^2\ g^{-1}$)	21.367	10.211
Total pore volume (cc g^{-1})	46.620 x 10^{-3}	10.057 x 10^{-3}
Average pore radius (Angstrom)	43.638	19.698

The surface area and porosity of rice straw ash and silica gel were analyzed using the BET surface area method which can be measured through nitrogen gas adsorption. Based on **Table 1**, the characteristics of silica gel are very different from rice straw ash. Based on the physical properties of its surface area, rice straw ash has a larger surface area compared to silica gel. This is due to the release of H_2O molecules. The large volume of rice straw ash is due to the presence of H_2O molecules of an amorphous form, thus filling the spaces in the material and making the pore volume wider. Meanwhile, silica gel is the result of drying in the oven, so that the pore volume shrinks.

CONCLUSION

Rice straw waste can be utilized to produce silica gel through sol-gel method. The rice straw-based silica gel obtained from calcination at 700^{o}C and leaching with NaOH has similar characteristics to general one. Based on IR analysis, siloxane active functional groups have been formed in sufficient numbers. Through crystallinity analysis, the silica gel has formed a crystal at 2θ = 31.71; 45,26; and 56.37 with high intensity. The surface area and pore volumes are 10.211 $m^2\ g^{-1}$ and 10.057x10^{-3} cc g^{-1}. This innovative approach contributes to the circular economy and underscores the importance of repurposing agricultural wastes to create valuable and eco-friendly products for the sustainability growth.

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REFERENCES

Abellan-Garcia, J., Martinez, D. M., Khan, M. I., Abbas, Y. M., & Pellicer-Martínez, F. (2023). Environmentally friendly use of rice husk ash and recycled glass waste to produce ultra-high-performance concrete. *Journal of Materials Research and Technology*, *25*, 1869–1881. https://doi.org/10.1016/J.JMRT.2023.06.041

Ali, & Asghar, H. (2010). Synthesis of quality silica gel; optimization of parameters. *Journal of Faculty of Engineering & Technology*, *16*(1), 19–32.http://111.68.103.26/journals/index.php/jfet/article/view/13

Bhattacharya, M., & Mandal, M. K. (2018). Synthesis of rice straw extracted nano-silica-composite membrane for CO2 separation. *Journal of Cleaner Production*, *186*, 241–252. https://doi.org/10.1016/J.JCLEPRO.2018.03.099

Badan Pusat Statistik. (2019). Statistics Sleman Regency. In *4 July 2019*. https://slemankab.bps.go.id/statictable/2019/07/04/455/luas-panen-produksi-dan-rata-rata-produksi-padi-sawah-dirinci-per-kecamatan-di-kabupaten-sleman-2017.html

Dharsono, Sumarno, & Chandra, N. R. A. (2019). Pemanfaatan jerami sebagai produk kreatif non industrial sebagai dasar pertimbangan desain. *Prosiding: Seni, Teknologi, dan Masyarakat*, *2*, 241–244. https://doi.org/10.33153/SEMHAS.V2I0.127

Ekwenna, E. B., Wang, Y., & Roskilly, A. (2023). The production of bio-silica from agro-industrial wastes leached and anaerobically digested rice straws. *Bioresource Technology Reports*, *22*, 101452. https://doi.org/10.1016/J.BITEB.2023.101452

Goodman, B. A. (2020). Utilization of waste straw and husks from rice production: A review. *Journal of Bioresources and Bioproducts*, *5*, 143–162. https://doi.org/10.1016/j.jobab.2020.07.001

Herdiana. (2020). Pemanfaatan limbah sekam padi dan jerami sebagai pupuk organik cair (POC) sebagai peluang usaha baru di desa Bonder kecamatan Praya Barat, NTB. *Avesina: Media Informasi Ilmiah Universitas Islam Al-Azhar*, *13*(02), 36–40. https://e-journal.unizar.ac.id/index.php/avesina/article/view/204

Hindryawati, N., & Alimuddin, A. (2010). Sintesis dan karakterisasi silika gel dari abu sekam padi dengan menggunakan natrium hidroksida (naoh). *Jurnal Kimia Mulawarman*, *7*(2),75–77. http://jurnal.kimia.fmipa.unmul.ac.id/index.php/JKM/article/view/1208

Karisma, K. (2015). *Pembuatan bioetanol dari jerami padi (Oryza sativa L) melalui proses sakarifikasi dan fermentasi serentak (SFS)*. Universitas Islam Negeri Alauddin Makassar.

Khorsand, H., Kiayee, N., Amir, &, Masoomparast, H., & Masoomparast, A. H. (2013). Optimization of amorphous silica nanoparticles synthesis from rice straw ash using design of experiments technique. *Particulate Science and Technology*, *31*(4),366–371.

https://doi.org/10.1080/02726351.2012.755587

Lu, P., & Hsieh, Y. Lo. (2012). Preparation and characterization of cellulose nanocrystals from rice straw. *Carbohydrate Polymers*, *87*(1), 564–573. https://doi.org/10.1016/J.CARBPOL.2011.08.022

Moraes, I. (2016). Erratum to: The next generation in membrane protein structure determination (Advances in experimental medicine and biology, 922, 10.1007/978-3-319-35072-1). In *Advances in Experimental Medicine and Biology* (Vol. 922). https://doi.org/10.1007/978-3-319-35072-1_13

Motlagh, E. K., Asasian-Kolur, N., & Sharifian, S. (2020). A comparative study on rice husk and rice straw as bioresources for production of carbonaceous adsorbent and silica. *Biomass Conversion and Biorefinery*, *12*(2022), 5729–5738. https://doi.org/10.1007/s13399-020-01145-7/Published

Niu, Y., Qu, R., Chen, H., Mu, L., Liu, X., Wang, T., Zhang, Y., & Sun, C. (2014). Synthesis of silica gel supported salicylaldehyde modified PAMAM dendrimers for the effective removal of Hg(II) from aqueous solution. *Journal of Hazardous Materials*, *278*, 267–278. https://doi.org/10.1016/J.JHAZMAT.2014.06.012

Nurmalasari, A. I., Supriyono, S., Budiastuti, M. T. S., Sulistyo, T. D., & Nyoto, S. (2021). Pengomposan jerami padi untuk pupuk organik dan pembuatan arang sekam sebagai media tanam dalam demplot kedelai. *PRIMA: Journal of Community Empowering and Services*, *5*(2), 102–109. https://doi.org/10.20961/prima.v5i2.44766

Pal, P., Li, H., & Saravanamurugan, S. (2022). Removal of lignin and silica from rice straw for enhanced accessibility of holocellulose for the production of high-value chemicals. *Bioresource Technology*, *Volume 361*, 127661. https://doi.org/https://doi.org/10.1016/j.biortech.2022.127661

Pandu, Mulyono, A., Purwaningrum, L., & Sulistyono, I. B. (2021). Analisa Karakter Material Jerami Padi Untuk Pemanfaatan Produk Kerajinan Tangan. *Widyakala Journal : Journal Of Pembangunan Jaya University*, *8*(2),97–103. https://doi.org/10.36262/widyakala.v8i2.509

Pillai, P., Dharaskar, S., Shah, M., & Sultania, R. (2020). Determination of fluoride removal using silica nano adsorbent modified by rice husk from water. *Groundwater for Sustainable Development*, *11*, 100423. https://doi.org/10.1016/J.GSD.2020.100423

Robles-Jimarez, H. R., Sanjuan-Navarro, L., Jornet-Martínez, N., Primaz, C. T., Teruel-Juanes, R., Molins-Legua, C., Ribes-Greus, A., & Campíns-Falcó, P. (2022). New silica based adsorbent material from rice straw and its in-flow application to nitrate reduction in waters: Process sustainability and scale-up possibilities. *Science of The Total Environment*, *805*, 150317. https://doi.org/https://doi.org/10.1016/j.scitotenv.2021.150317

Rusdianasari, R., Taufik, M., Bow, Y., & Fitria, M. S. (2020). Application of Nanosilica from Rice Husk Ash as Iron Metal (Fe) Adsorbent in Textile Wastewater. *Indonesian Journal of Fundamental and Applied Chemistry*, *5*(1),7–12. https://ijfac.unsri.ac.id/index.php/ijfac/article/view/167/87

Said, A., Fatimah, I., & Rubiyanto, D.

(2014). Effect of temperature on rice husk asing and its application on adsorbing fe and zn metal in patchouli oil. *EKSAKTA: Journal of Sciences and Data Analysis*, *14*(2), 71–86. https://doi.org/10.20885/EKSAKTA.VOL14.ISS2.ART7

Sudhan, N., Subramani, K., Karnan, M., Ilayaraja, N., & Sathish, M. (2017). Biomass-derived activated porous carbon from rice straw for a high-energy symmetric supercapacitor in aqueous and nonaqueous electrolytes. *Energy and Fuels*, *31*(1), 977–985. https://doi.org/10.1021/ACS.ENERGYFUELS.6B01829/ASSET/IMAGES/LARGE/EF-2016-01829G_0006.JPEG

Sugama, I. N., & Budiari, N. L. G. (2012). Pemanfaatan jerami padi sebagai pakan alternatif untuk sapi bali dara. *Majalah Ilmiah Peternakan*, *15*(1), 21–25. https://doi.org/10.24843/MIP.2012.V15.I01.P04

Winarsih, S. (2013). *Pemanfaatan jerami padi untuk produksi bioetanol dengan pretreament microwave alkali dan hidrolisis menggunakan enzim kasar dari Trichoderma reesei dan Aspergillus niger*. Universitas Brawijaya.

Yekeen, N., Salampessy, S. N., Abu Bakar, A. H., Ali, M., Okunade, O. A., Musa, S. A., & Bavoh, C. B. (2023). Synthesis and pore-scale visualization studies of enhanced oil recovery mechanisms of rice straw silica nanoparticles. *Geoenergy Science and Engineering*, *221*, 111292. https://doi.org/10.1016/J.PETROL.2022.111292